THE RHEOLOGICAL STUDY OF SLURRY CONTAINING CHINA CLAY AND BALL CLAY USING DIFFERENT DEFLOCCULANT

A thesis submitted in the partial fulfillment of the requirements for the degree of Bachelor of Technology

By

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CERTIFICATE

This is to certify that the thesis entitled, "The Rheological Study Of Slurry Containing China Clay And Ball Clay Using Different Deflocculant" submitted by Siddhartha Ranjan Behera (110CR0095) for partial fulfillments of the requirements for the award of Bachelor of Technology degree in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied in this thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date

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Siddhartha Ranjan Behera

B.Tech Ceramic Engineering

CONTENTS

ABSTRACT

Different proportion of china clay and ball clay were taken to make clay mixture containing china clay: ball clay in the ratio 70:30, 80:20, 90:10. The clay mixture were used to prepare clay slurries with different solid loading viz. 15, 20 and 25 vol%. Three

Deflocculants e.g. Sodium Silicate, Darvan C and Sodium Carbonate were taken in the proportion 0.2%, 0.4% and 0.6% and the clay slurries were stabilized by pot milling for 10 hours. Shear stress- Shear rate behavior, viscosity- shear rate variation, τ_{gel},τ_{yeild} were studied as a function of deflocculant type, amount and solid loading. Settling heights and pH change of the slurries were also studied. The study indicates that the optimized slurry should have 20% solid loading and 0.2% Deflocculant.

CHAPTER 1

INTRODUCTION

Rheology is the science of deformation and flow. The knowledge of rheological behavior is very essential for the selection and designing of equipment for storing, pumping, transporting, milling, atomizing and forming a ceramic system. Rheological study is inevitable for the research and development of the slurry system.[1] The rheological properties of slip during slip casting is very important as it is a main parameter which controls the flow behavior during casting, settling behavior and properties of casting. Ceramic slurries and pastes are relatively complex and poorly characterized. The interparticle spacing depends widely on the solid loading, the state of dispersion and the particle packing. The rheological property of the slip depends on the physical and chemical property of the raw material and the conditions under which the slip is prepared. So depending on the place of production of raw material the rheological properties changes. The rheological properties of the slip affect properties of cast behavior, casting time etc. The important parameters which affect the rheological behavior of the slurry are chemical and mineralogical composition of raw materials, viscosity, particle shape and size, temperature, time, type of mixing, pH of slurry. During slurry preparation, modification of particular parameter is done to achieve desired properties. Slip viscosity effectively controls the properties of ceramic casting slips. The quality and thickness of the cake formed can be changed by changing the slip viscosity. The water used for the slip casting process must be free from ions which effects the colloidal properties of the particles present in the suspension. As with change in concentration of these ion, the rheological properties of the slurry changes depending whether it does a flocculating or deflocculating action. [2]

China clay and ball clay are the two raw materials which are easily available and economic. Hence the study of rheology of china clay and ball clay is more reasonable and cost effective. Moreover, china clay and ball clay are used as a basic material for ceramic slurry preparation. Hence their rheology should be studied as it helps in deciding many primary ceramic industrial application.

Deflocculant is a chemical additive used to reduce the viscosity of the suspension. It prevents the flocculation of the suspended particles. Deflocculant are substances which prevent flocculation by increasing zeta potential and therefore the repulsive forces between particles. The electrical potential between the surface and the bulk of the solution drops gradually away from the surface across a distance called double layer. The potential at the slippage plane, somewhere in between the double layer is called zeta potential. [1]

The following paragraphs discuss some of the common deflocculants, their properties and applications:

(a) $Na_2O.nSiO_2$ (sodium silicate)

(b) It is one of the most effective and reliable deflocculant used in slip casting of different slurries .It is cheap and easily available which increases its demand. The ratio of SiO_2 to Na_2O in sodium silicate can vary from 3.75:1 to 1:1 and is available in liquid or solid form. Sodium silicate increases the pH of the suspension, due to hydrolysis and the silicon separates out in the form of colloidal silica which performs as a protective colloid, according to the following reaction:

$$Na_2O.nSiO_2 + H_2O \longrightarrow nSiO_2 + 2Na^+ + 2OH^-$$

(c) <u>Darvan C</u>

Darvan C is an ammonium polymethacrylate stabilizer. It has low ash content and works effectively when prolonged ball milling or shear mixing is done. It is a well-known deflocculant which is used to disperse ceramic suspension and hence minimize the water content. It is the liquid alternative to the long popular sodium silicate. It has various advantages like nonreactive to plaster mold, less sensitive to localized variation in quantity and hence recyclable, stable during prolonged milling, low ash content, small addition provides high fluidity and slip stability [3]

CHAPTER 2

LITERATURE REVIEW

Adriano Michael Bernardin et al. [4]studied of the rheological behavior of porcelain tile slurries produced by wet milling. They analyzed the deflocculation of slurries using two different types of deflocculants (sodium meta silicate and sodium silicate). The slurry rheology was studied by observing the change in viscosity as a function of deflocculant. After the analysis they concluded that sodium silicate is more effective than sodium meta silicate. The best results for silicate were found at SiO_2/Na_2O ratio 2:1 and that of metasilicate 3:1. This study shows the importance of viscosity control of samples in ceramic industry. They also found to know that the rheological behaviors of the ceramic suspensions are not only effected by the deflocculant taken but also by the hardness of water and pH and different characteristic of particles e.g.:- shape, particle size.

Himanshu Desai [5]studied the rheological behavior of clay water system with different surfactants, viz.nonionic triton X-100 (TX -100), nonionic sodium dodecyl benzene sufonate (SDBS) and cationic cetyl pyridinium bromide (CPB) surfactant on pyrophilite –water slurryusing rotational cone and plate BOHLIN – VISCO 88 viscometer. It was observed that the addition of cationic surfactant to pyrophilite slurry caused an increase in viscosity and then a decrease in viscosity due to charge reversal. However, in case of nonionic surfactant the viscosity first increased after which it remained constant.

F.N. Shi et al.[6]found out a new procedure for obtaining full shear stress- shear rate flow rate curve for different unstable slurries using the single bobbin- Debex on-line viscometer. They showed that torque coefficient data from a variety of Newtonian fluids and non-Newtonian slurries fell on a single curve, which was characteristic for a particular instrument design.

C. M.Gomes et al. [7]studied the effect of two different type of sodium silicate on rheological behavior of triaxial ceramic suspension with 40 wt% solid loading for both the composition and determined the minimum amount of deflocculant required for stabilizing the slurry. It was noted that an increase in alkalinity of the sodium silicate resulted in lowering of viscosity of the dispersion.

R.R. Klimpel [8] found out that wet grinding of materials in most industrial grinding devices can be significantly changed by slurry rheology. He concluded that pulp density, the level of fineness present and pulp chemistry are important to maximize the throughput.

F.H. Norton [9] carried out research on specially samples prepared from natural clay. The study was made on kaolinite samples with known particle size, known organic content and known controlled adsorbed ions. The effect of any one variable on the plasticity and that workability of clay slip were studied. On basic information water clay system is needed to understand the complex behavior of natural clays.

E.-Jen Teh et al. [10] studied the effect of kaolinite clays sources having similar mineral composition on rheological and electrokinetic behavior. The difference in behavior was attributed to the difference in surface chemistry of the clay particle. The edge chemistry of similar absorbing a small anionic additive such as citrate, produced a yield stress- pH behavior for the different kaolin clay slurries having similar characteristics. The yield stress behavior is due to the attraction between negatively charged faces of the clay and positively charged edges.

Lavanya Avadiar et al.[11]studied the effects of polyethlenimine dosages and molecular weights on flocculation, rheology and consolidation behaviors of kaolin slurries. They concluded that at high PEI dosages (polyethlenimine) the consolidation behaviors of kaolin slurries were different.

Chul-Woo Chung et al.[12]studied the flow behavior of cementitious slurry containing iron oxide and observed that the workability influences performance and preparation of cementitious form monolith.

CHAPTER 3

EXPERIMENTAL WORK

3.1 Clay slurry preparation

Clay slurries were prepared by mixing china clay and ball clay in different weight ratio so as to achieve solid loading of 15,20and 25 volume percent in the slurry. Three different deflocculants viz. Sodium Silicate, Darvan C, Sodium Carbonate were used for stabilizing the slurry. Each type of deflocculants were added either 0.2,0.4, 0.6 volume percent. Each mixture composition was wet milled in pot mill for 12hours using alumina grinding media. The volume of each slurry was 100ml.

Table.1- Batch composition for clay slurry with Sodium Silicate as deflocculant

China clay: Ball clay	Solid loading	Deflocculant(%)	Water (ml)
70:30	15	0.2	85
		0.4	
		0.6	
	20	0.2	80
		0.4	
		0.6	
	25	0.2	75
		0.4	
		0.6	
80:20	15	0.2	85
		0.4	
		0.6	
	20	0.2	80
		0.4	
		0.6	
	25	0.2	75
		0.4	
		0.6	
90:10	15	0.2	85
		0.4	
		0.6	
	20	0.2	80
		0.4	
		0.6	
	25	0.2	75
		0.4	
		0.6	

Table.2- Batch composition for clay slurry with Darvan C as deflocculant

China Clay: Ball Clay	Solid loading	Deflocculant(%)	Water (ml)
70:30	15	0.2	85
		0.4	
		0.6	
	20	0.2	80
		0.4	
		0.6	
	25	0.2	75
		0.4	
		0.6	
80:20	15	0.2	85
		0.4	
		0.6	
	20	0.2	80
		0.4	
		0.6	
	25	0.2	75
		0.4	
		0.6	
90:10	15	0.2	85
		0.4	
		0.6	
	20	0.2	80
		0.4	
		0.6	
	25	0.2	75
		0.4	
		0.6	

Table.3- Batch composition for clay slurry with Sodium Carbonate as deflocculant

<table>
<tr><th>Solid loading</th><th>Ball clay</th><th>China clay</th><th>Deflocculant(%)</th><th>Water(ml)</th></tr>
<tr><td rowspan="3">25</td><td rowspan="3">19.5</td><td rowspan="3">45.5</td><td>0.3</td><td rowspan="3">75</td></tr>
<tr><td>0.4</td></tr>
<tr><td>0.5</td></tr>
</table>

3.2Slurry Characterization

All the prepared clay slurries were tested for Shear – Stress Rate Behavior both during upswing and downswing, viscosity, settling height with time, pH change with time.

The slurries were subjected shear stress in a concentric Cylinder Rheometer Model Rheolab, Anton Parr at shear rates1-100 (sec^{-1}) and the shear stress and viscosity was noted.
The measurement was done both during upswing (i.e. 1- 100sec^{-1}) and down swing (100-1sec^{-1}) mode.

From the obtained graph τ_{gel} or Gelation stress was also obtained.

3.3 Measurement of Settling Height

Post rheology test, the slurries were allowed to stand for 24 hours and the settling height was measured.

3.4 pH change of the slurries

The change in pH of the slurries were measured for all the slurry composition using pH meter (Eutech pH tutor)

CHAPTER 4

RESULTS AND DISCUSSION

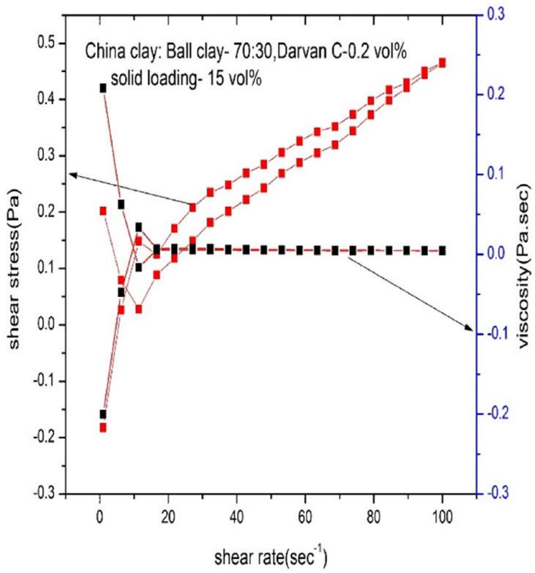


Figure 1: Variation of Shear Stress and Viscosity with Shear rate

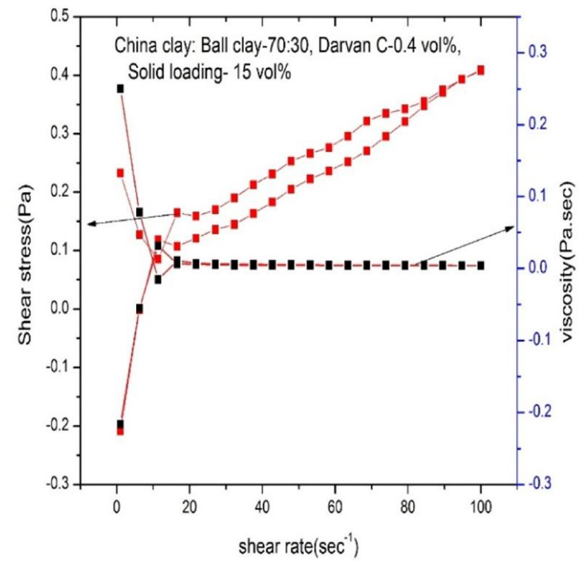


Figure 2: Variation of Shear Stress and Viscosity with Shear rate

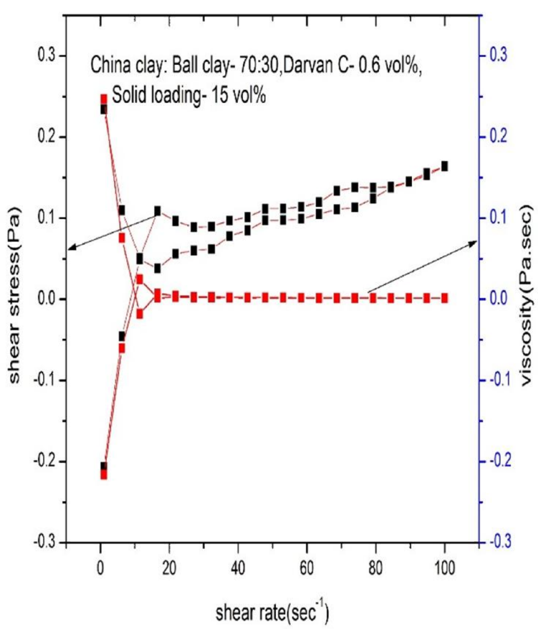


Figure 3: Variation of Shear Stress and Viscosity with Shear rate

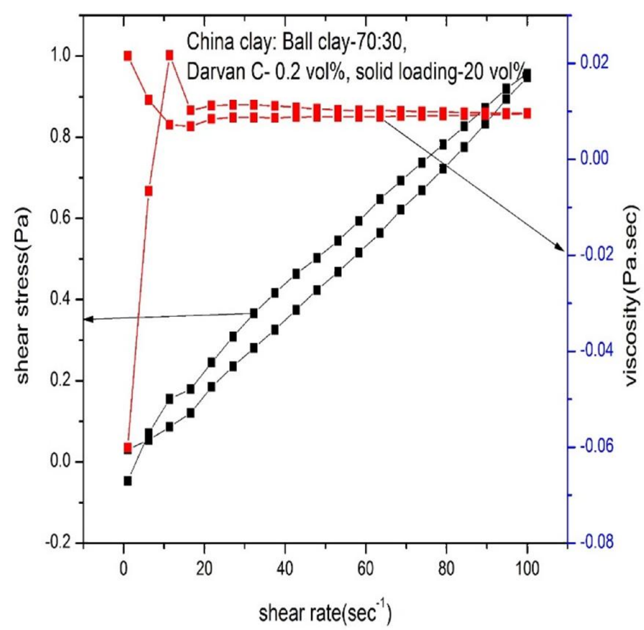
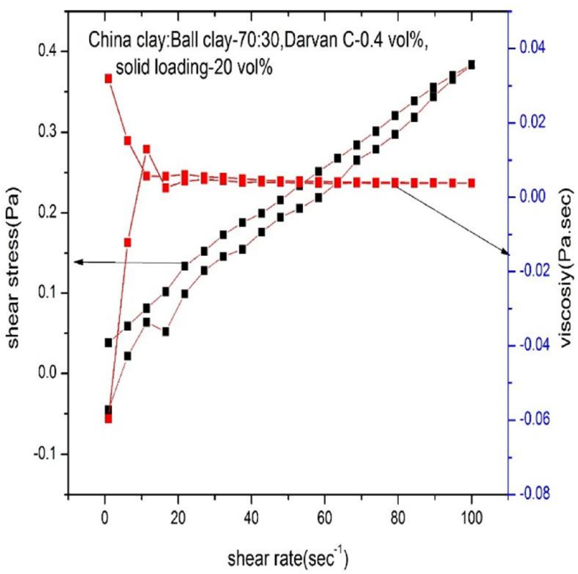


Figure 4: Variation of Shear Stress and Viscosity with Shear rate



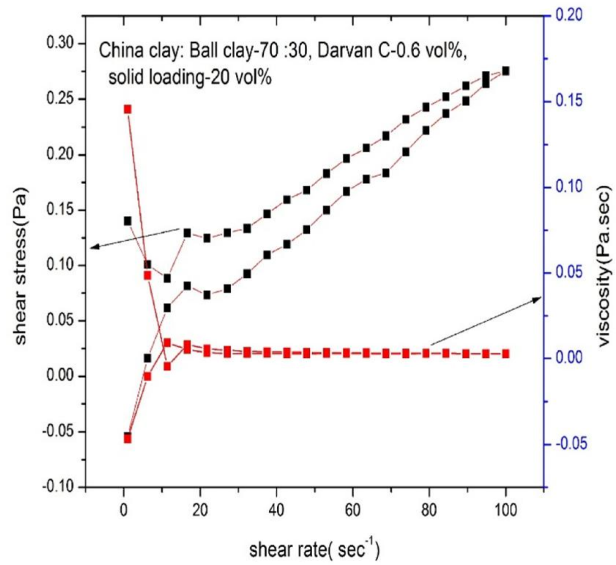
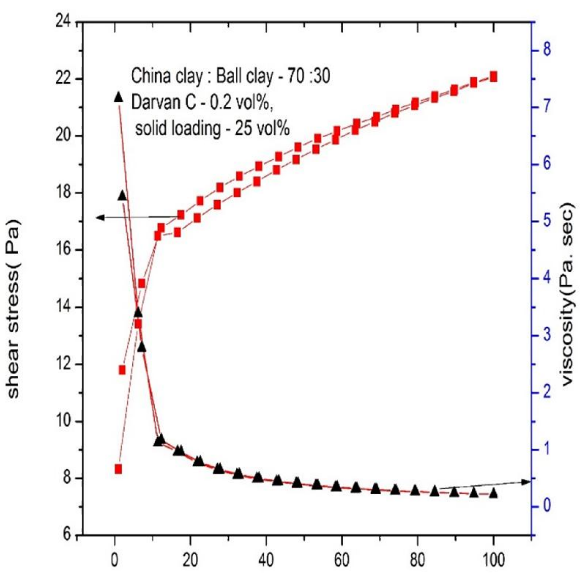


Figure 5: Variation of Shear Stress and Viscosity with Shear rate **Figure 6: Variation of Shear Stress and Viscosity with Shear rate**



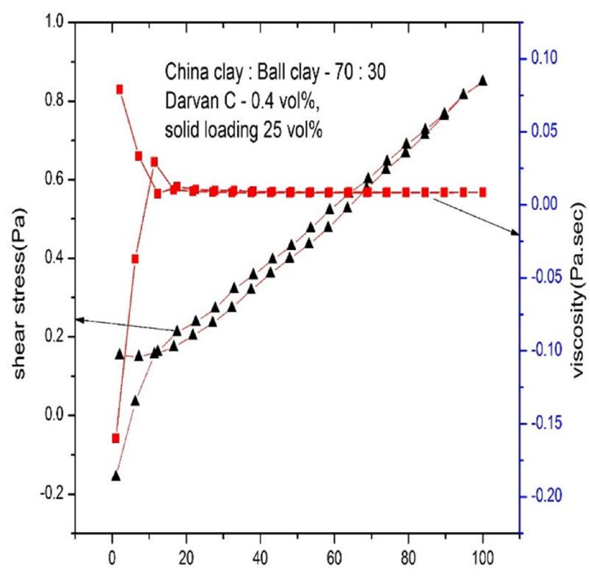
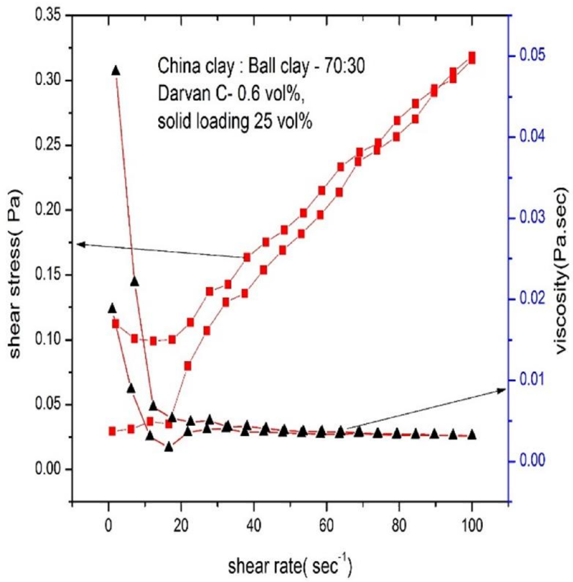


Figure 7: Variation of Shear Stress and Viscosity with Shear rate **Figure 8: Variation of Shear Stress and Viscosity with Shear rate**



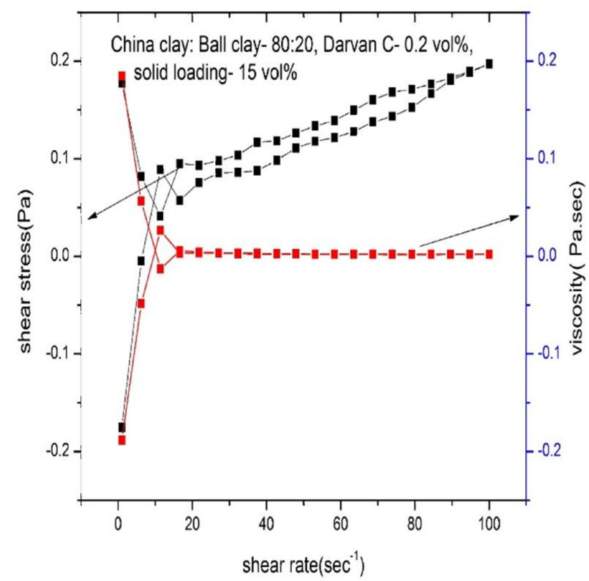
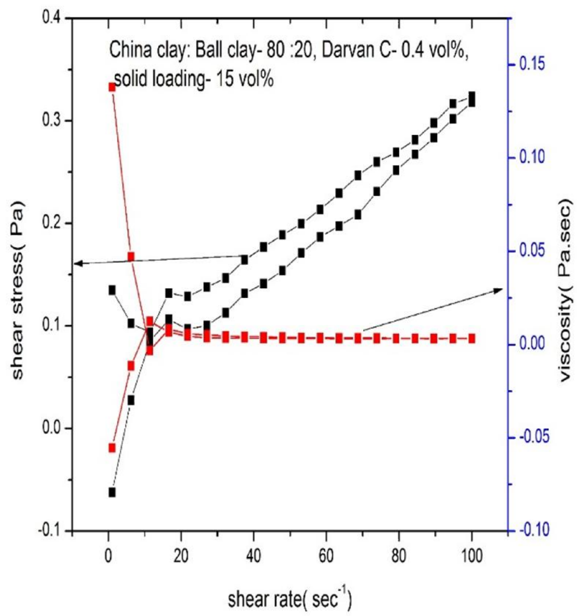


Figure 9: Variation of Shear Stress and Viscosity with Shear rate **Figure 10: Variation of Shear Stress and Viscosity with Shear rate**



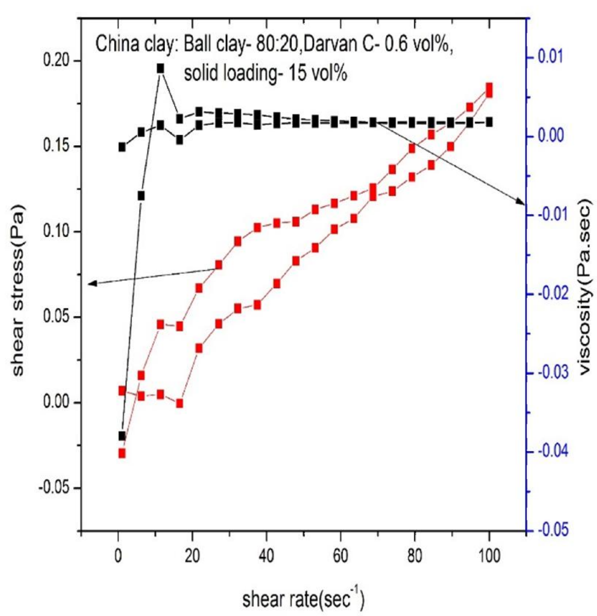
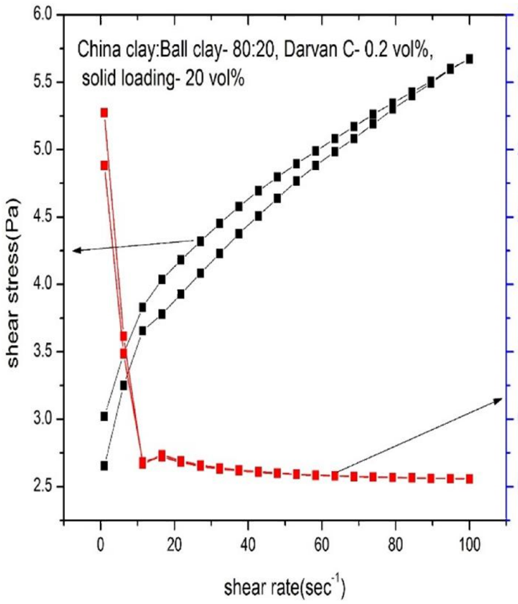


Figure 11: Variation of Shear Stress and Viscosity with Shear rate **Figure 12: Variation of Shear Stress and Viscosity with Shear rate**



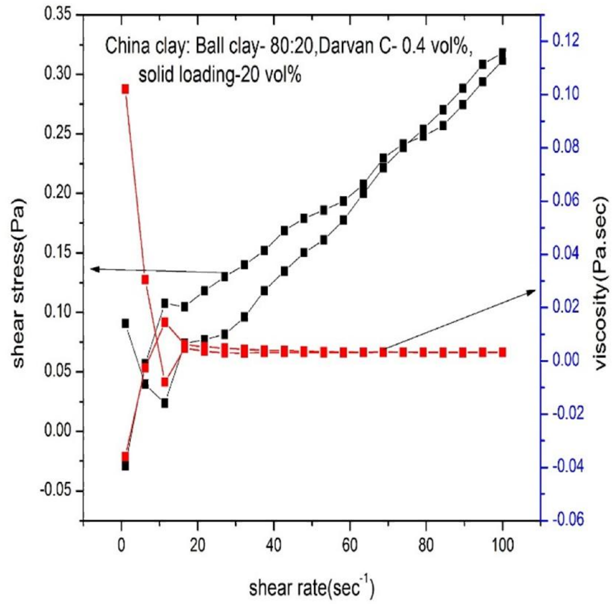
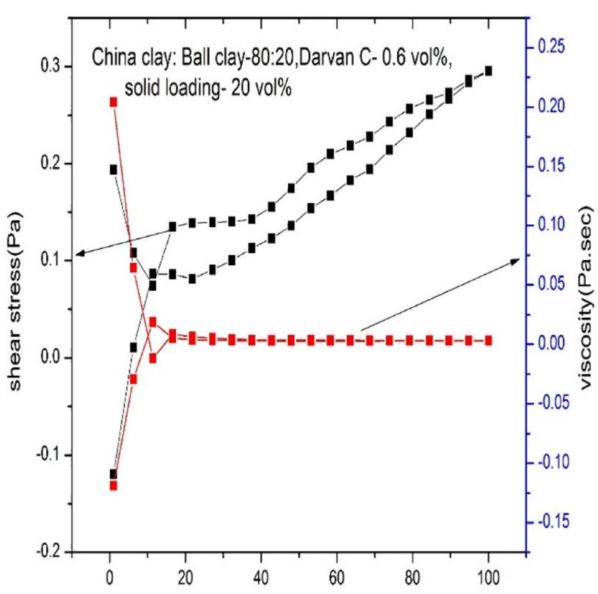


Figure 13: Variation of Shear Stress and Viscosity with Shear rate

Figure 14: Variation of Shear Stress and Viscosity with Shear rate



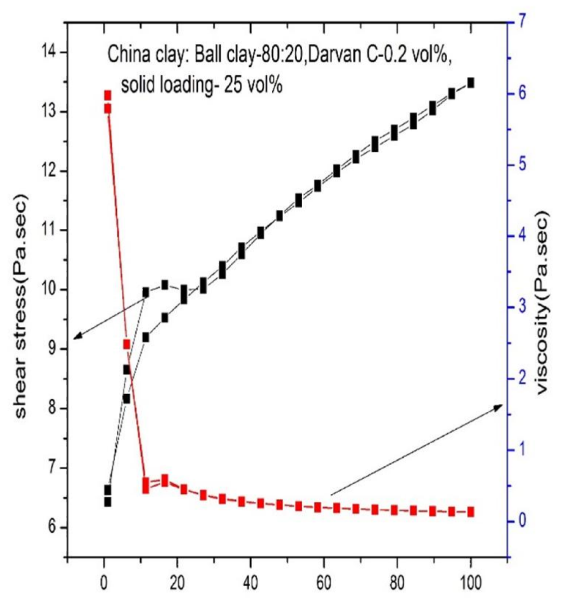
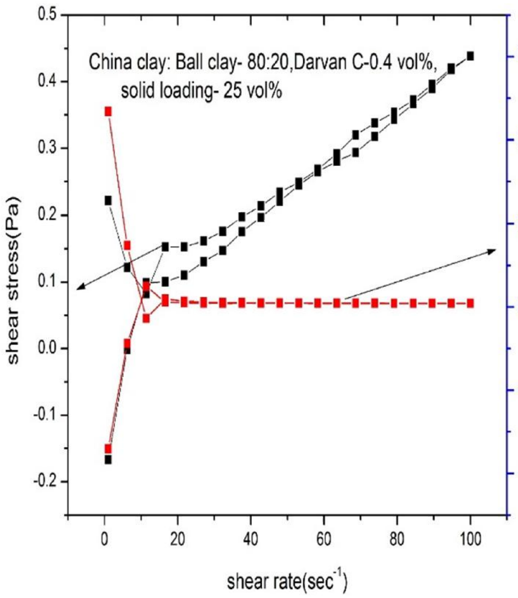


Figure 15: Variation of Shear Stress and Viscosity with Shear rate

Figure 16: Variation of Shear Stress and Viscosity with Shear rate



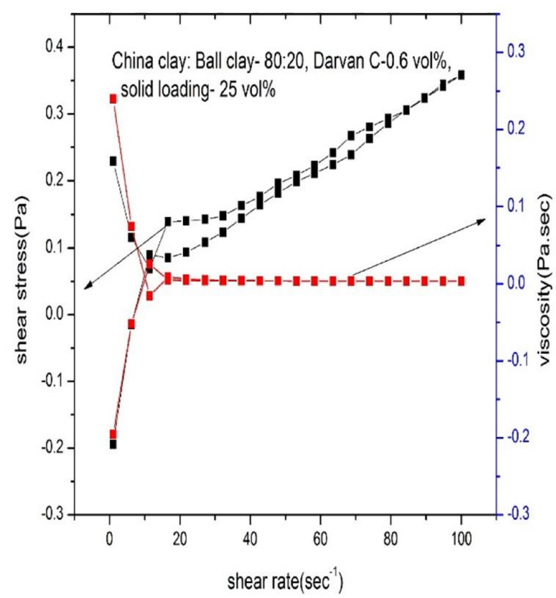
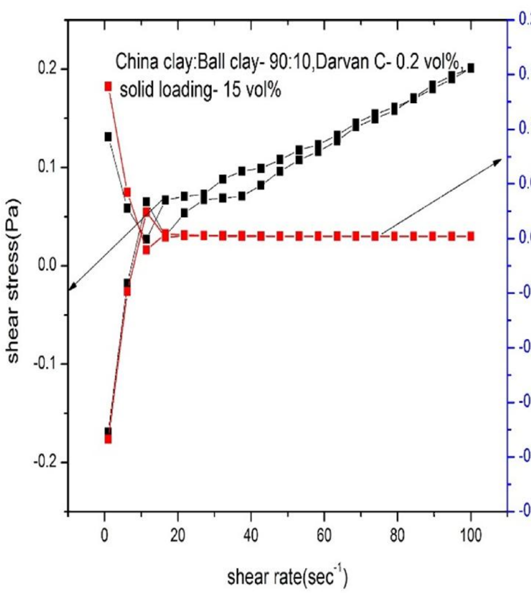


Figure 17: Variation of Shear Stress and Viscosity with Shear rate

Figure 18: Variation of Shear Stress and Viscosity with Shear rate



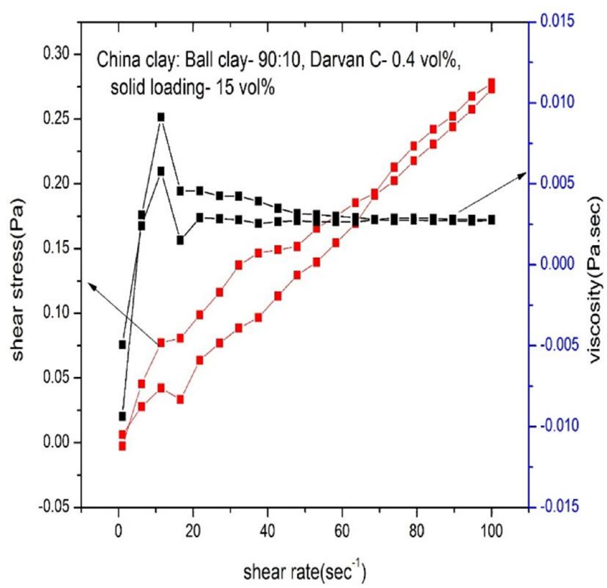
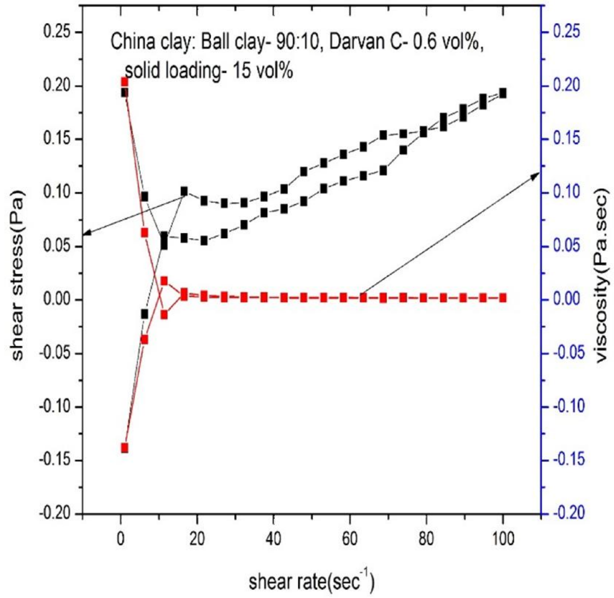


Figure 19: Variation of Shear Stress and Viscosity with Shear rate

Figure 20: Variation of Shear Stress and Viscosity with Shear rate



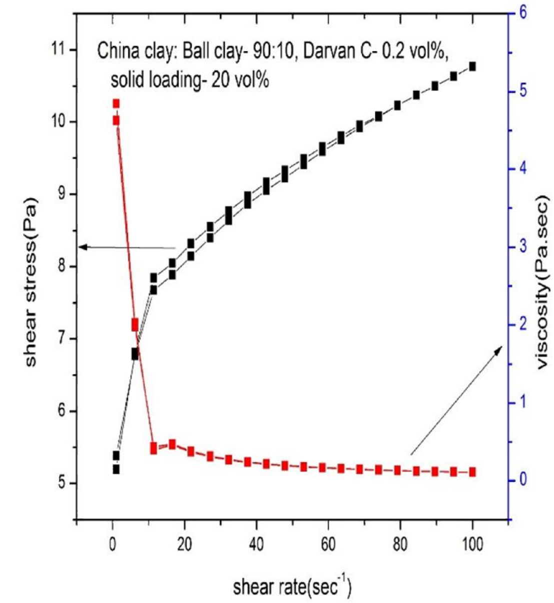
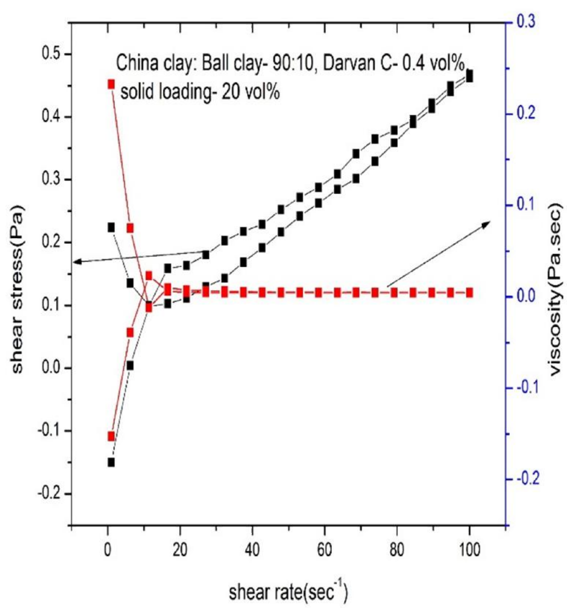


Figure 21: Variation of Shear Stress and Viscosity with Shear rate

Figure 22: Variation of Shear Stress and Viscosity with Shear rate



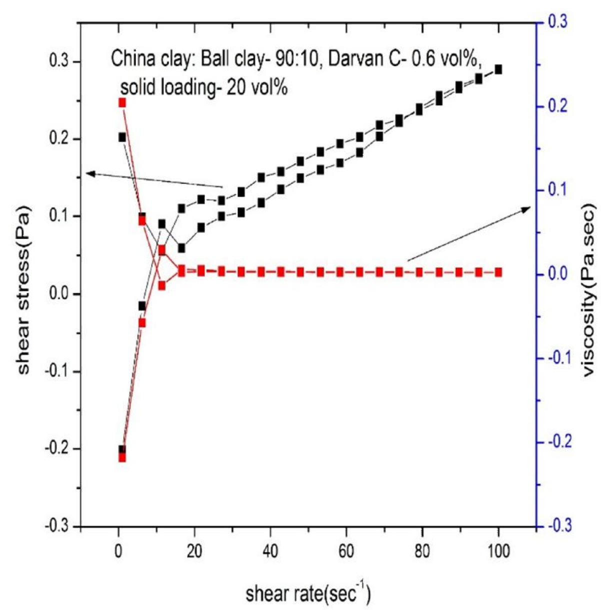
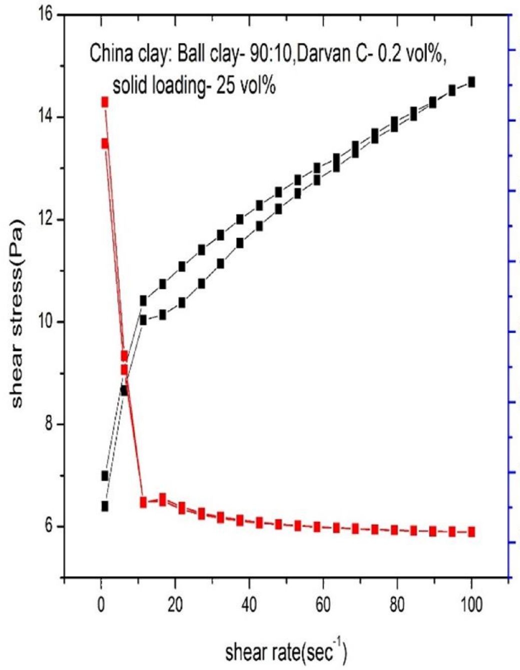


Figure 23: Variation of Shear Stress and Viscosity with Shear rate

Figure 24: Variation of Shear Stress and Viscosity with Shear rate



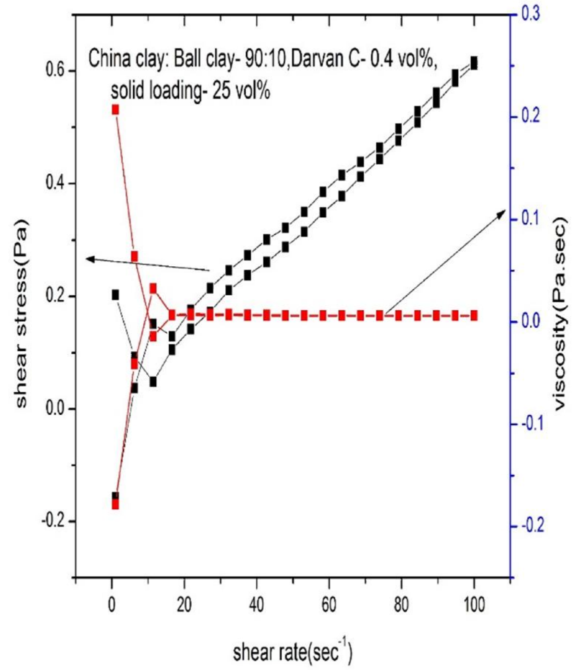
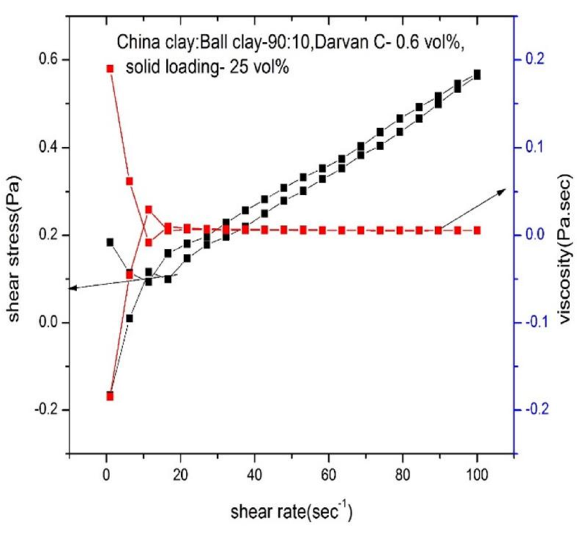


Figure 25: Variation of Shear Stress and Viscosity with Shear rate **Figure 26: Variation of Shear Stress and Viscosity with Shear rate**



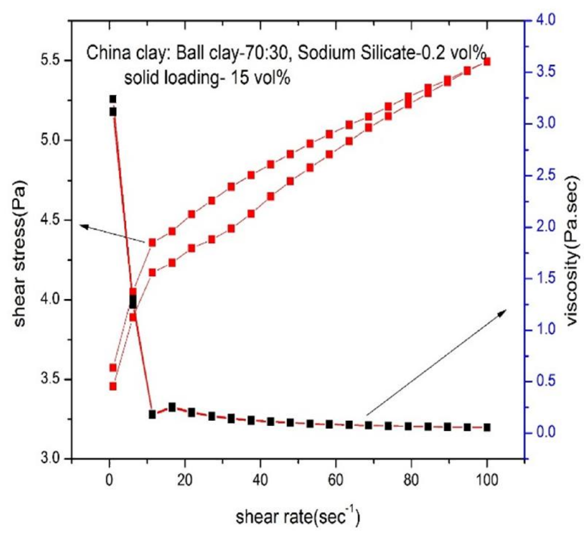
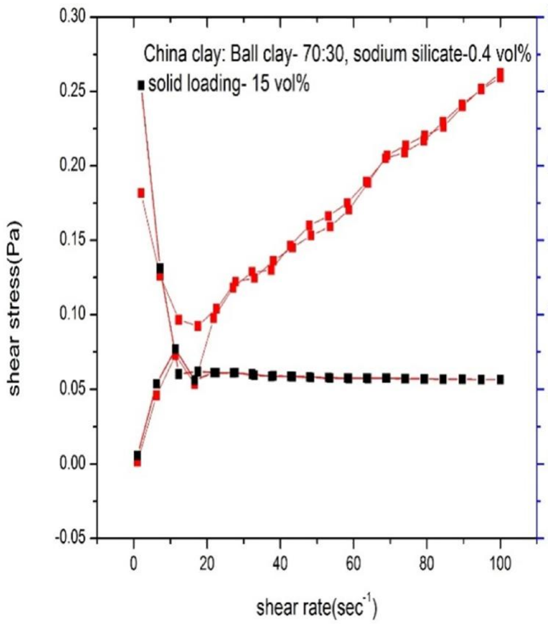


Figure 27: Variation of Shear Stress and Viscosity with Shear rate **Figure 28: Variation of Shear Stress and Viscosity with Shear rate**



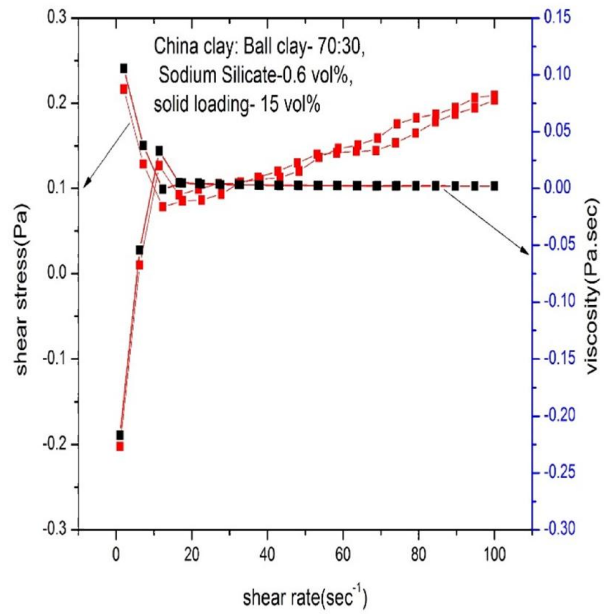
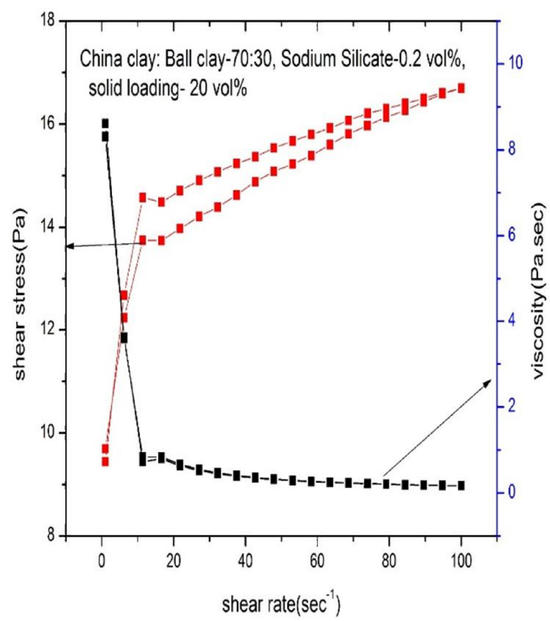


Figure 29: Variation of Shear Stress and Viscosity with Shear rate

Figure 30: Variation of Shear Stress and Viscosity with Shear rate



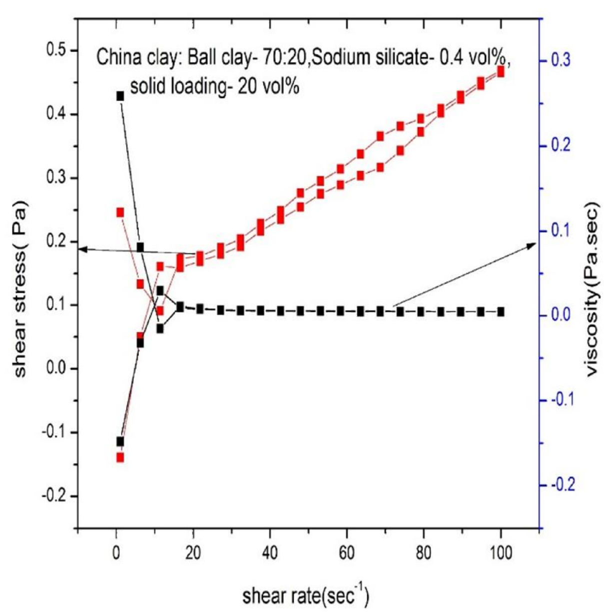


Figure 31: Variation of Shear Stress and Viscosity with Shear rate

Figure 32: Variation of Shear Stress and Viscosity with Shear rate

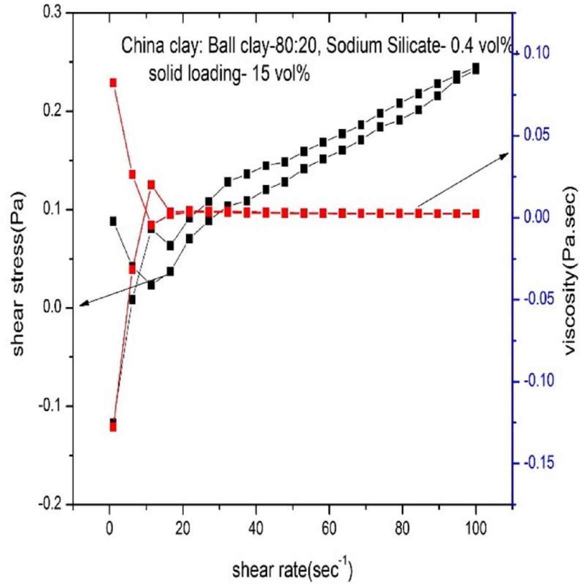


Figure 33: Variation of Shear Stress and Viscosity with Shear rate

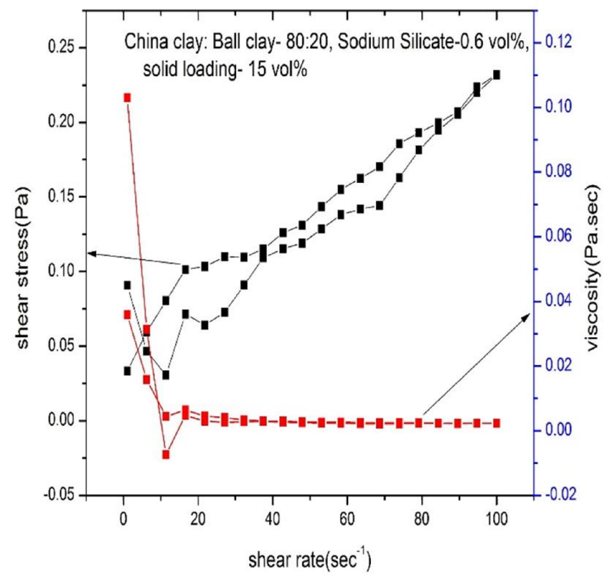


Figure 34: Variation of Shear Stress and Viscosity with Shear rate

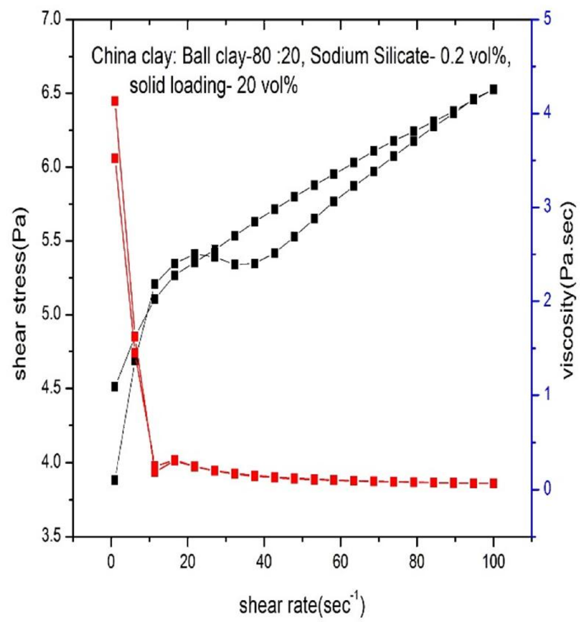


Figure 35: Variation of Shear Stress and Viscosity with Shear rate

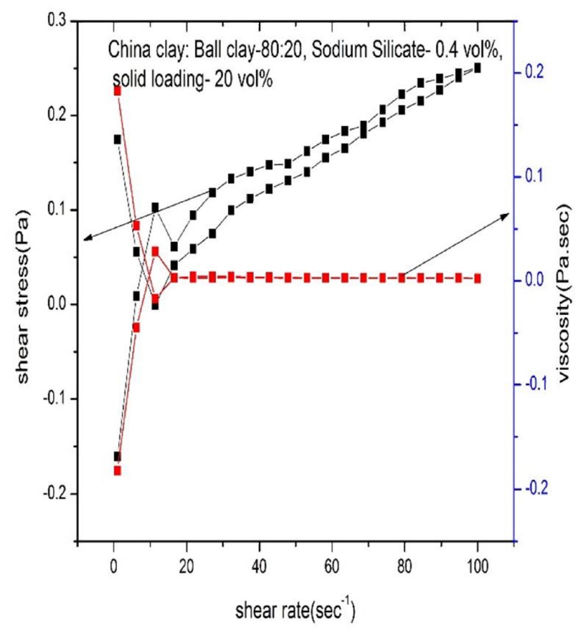
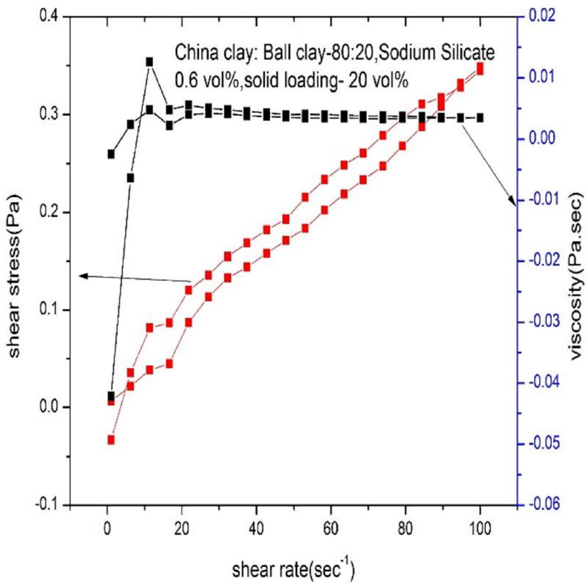


Figure 36: Variation of Shear Stress and Viscosity with Shear rate



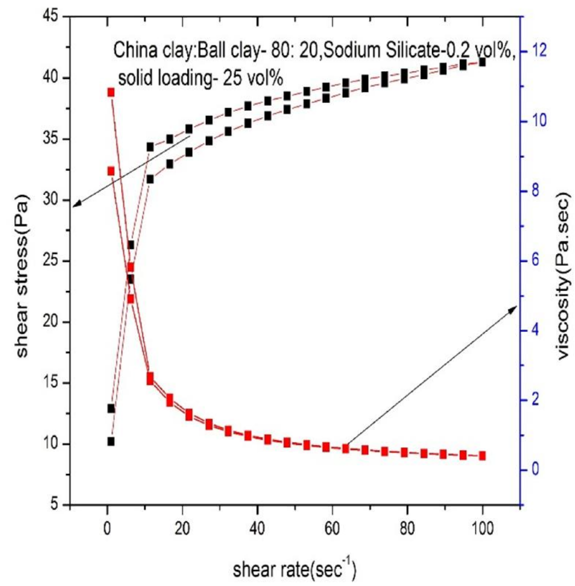
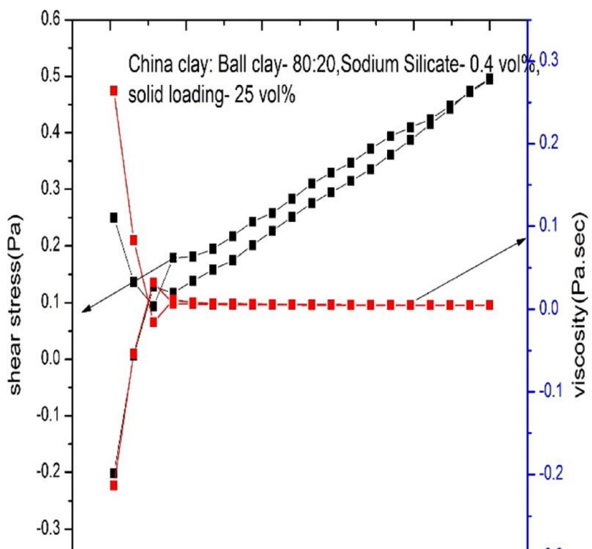


Figure 37: Variation of Shear Stress and Viscosity with Shear rate

Figure 38: Variation of Shear Stress and Viscosity with Shear rate



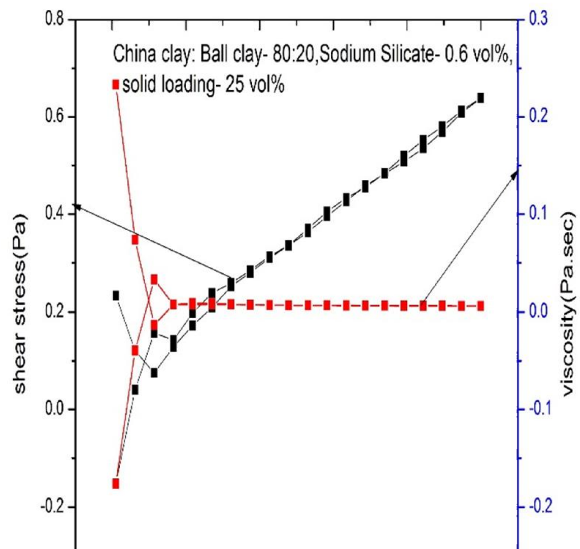


Figure 39: Variation of Shear Stress and Viscosity with Shear rate

Figure 40: Variation of Shear Stress and Viscosity with Shear rate

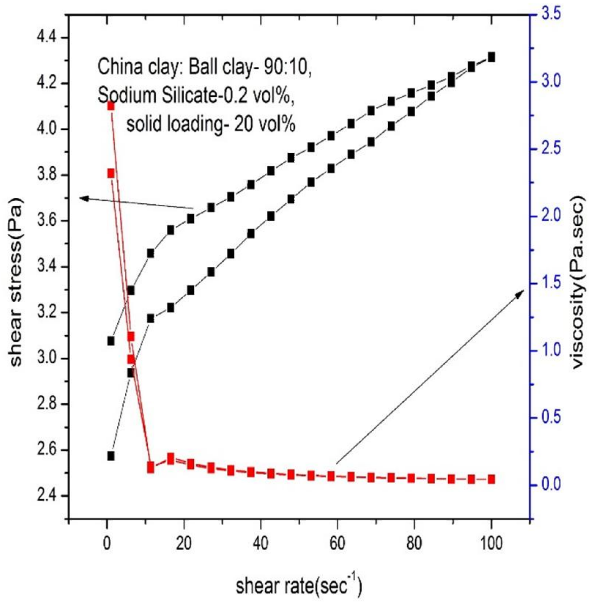


Figure 41: Variation of Shear Stress and Viscosity with Shear rate

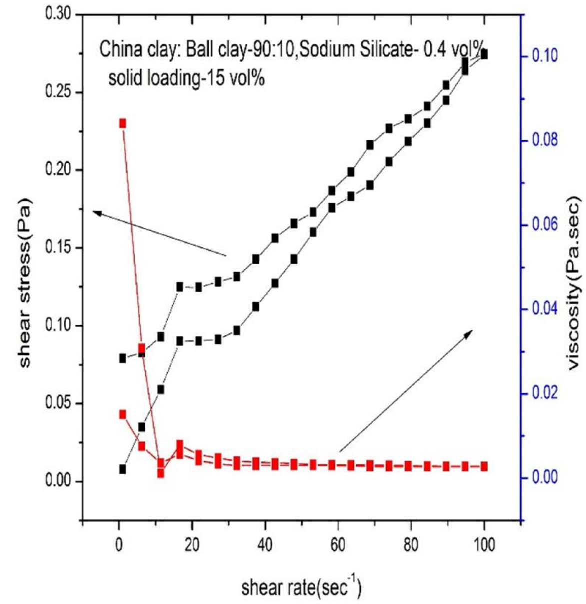


Figure 42: Variation of Shear Stress and Viscosity with Shear rate

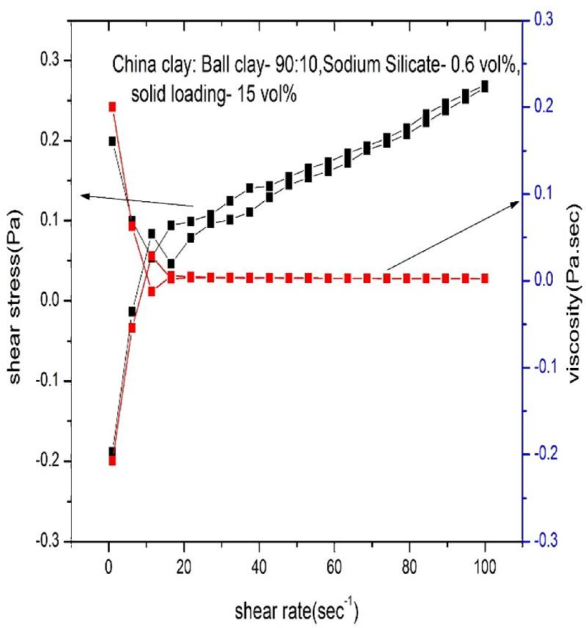


Figure 43: Variation of Shear Stress and Viscosity with Shear rate

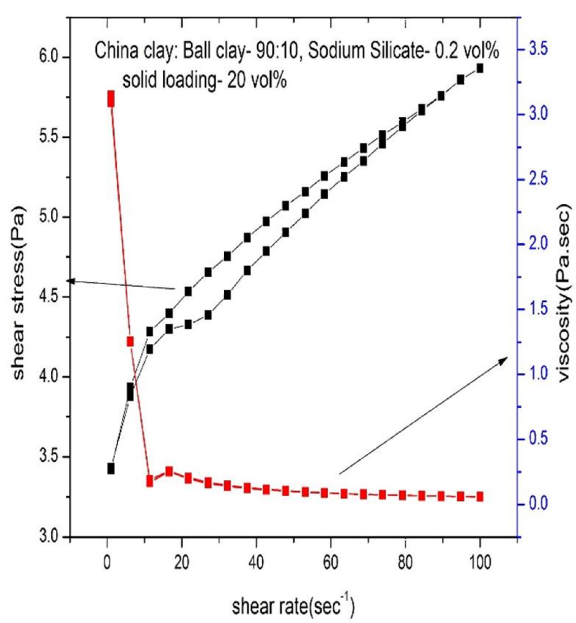


Figure 44: Variation of Shear Stress and Viscosity with Shear rate

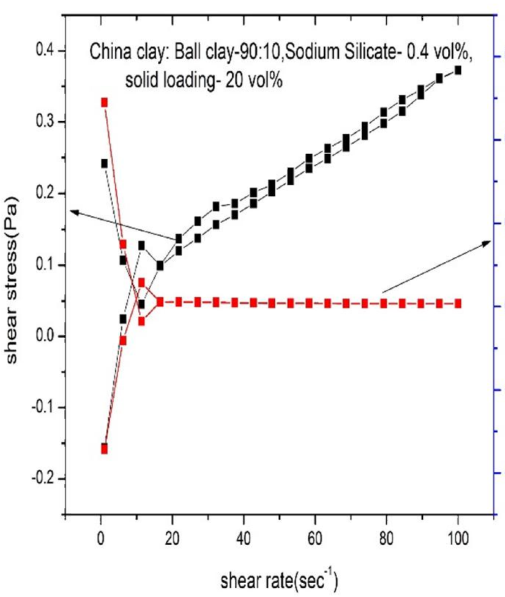


Figure 45: Variation of Shear Stress and Viscosity with Shear rate

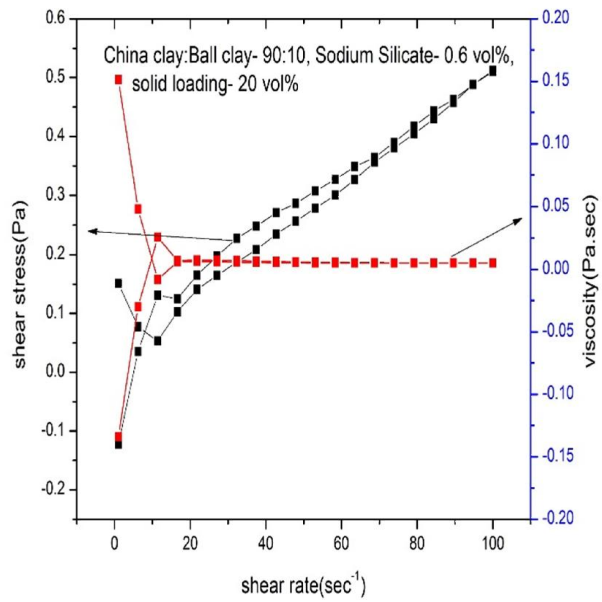


Figure 46: Variation of Shear Stress and Viscosity with Shear rate

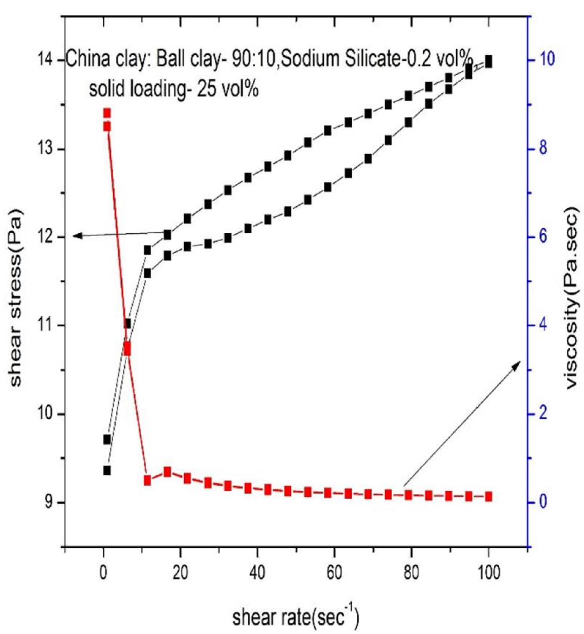


Figure 47: Variation of Shear Stress and Viscosity with Shear rate

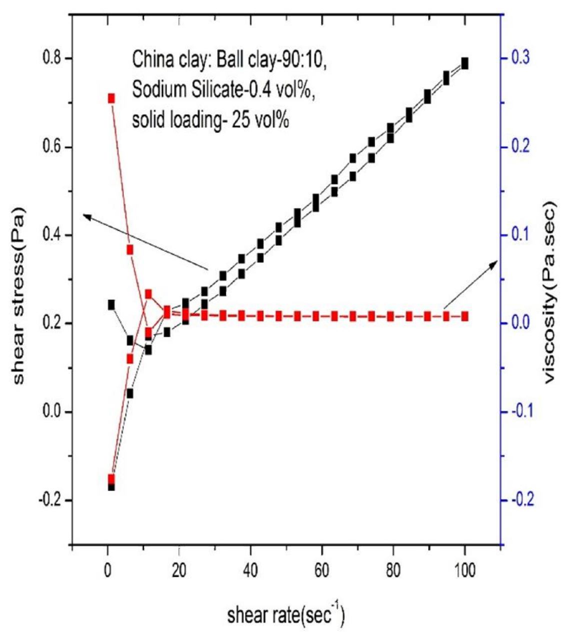
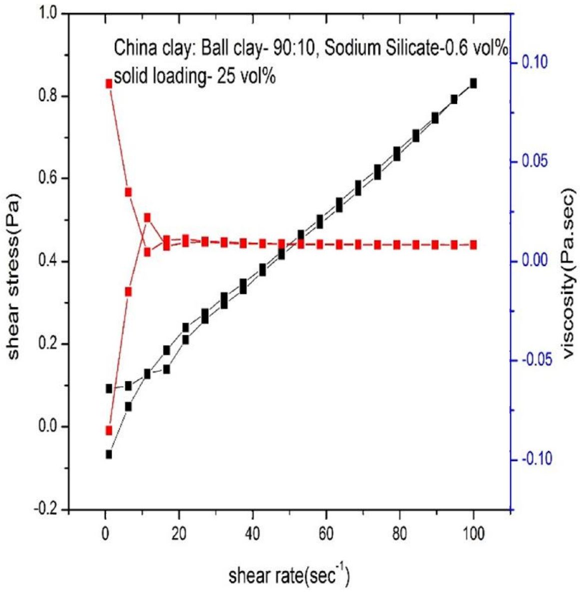


Figure 48: Variation of Shear Stress and Viscosity with Shear rate



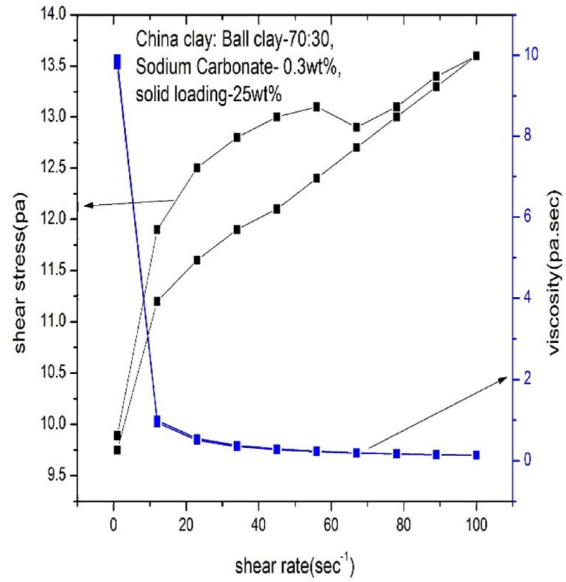
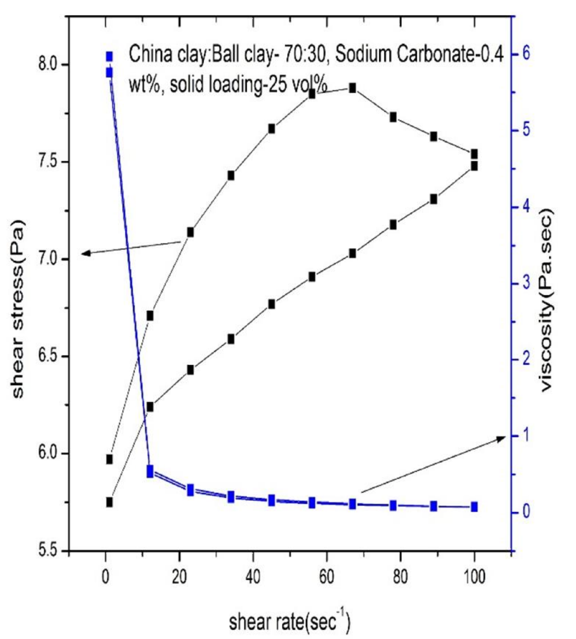


Figure 49: Variation of Shear Stress and Viscosity with Shear rate

Figure 50: Variation of Shear Stress and Viscosity with Shear rate



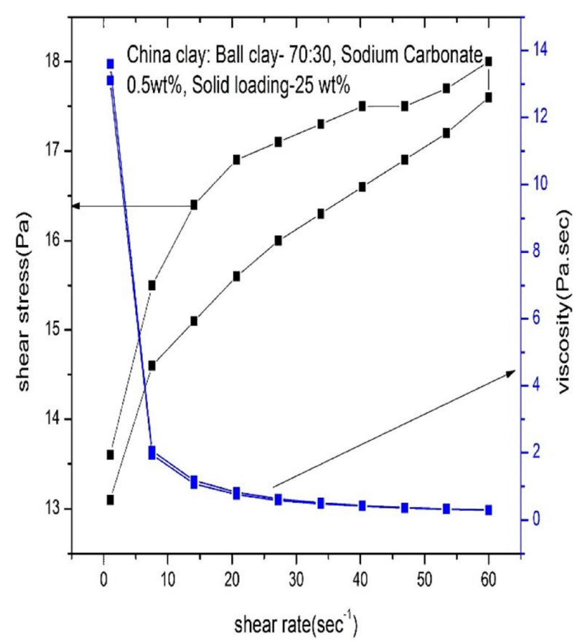
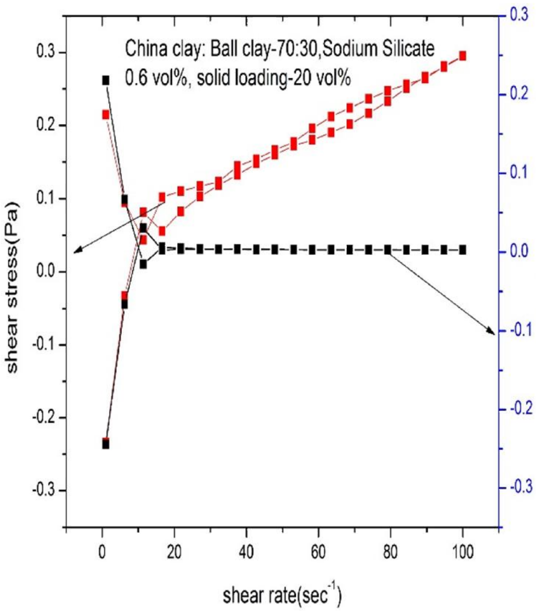


Figure 51: Variation of Shear Stress and Viscosity with Shear rate

Figure 52: Variation of Shear Stress and Viscosity with Shear rate



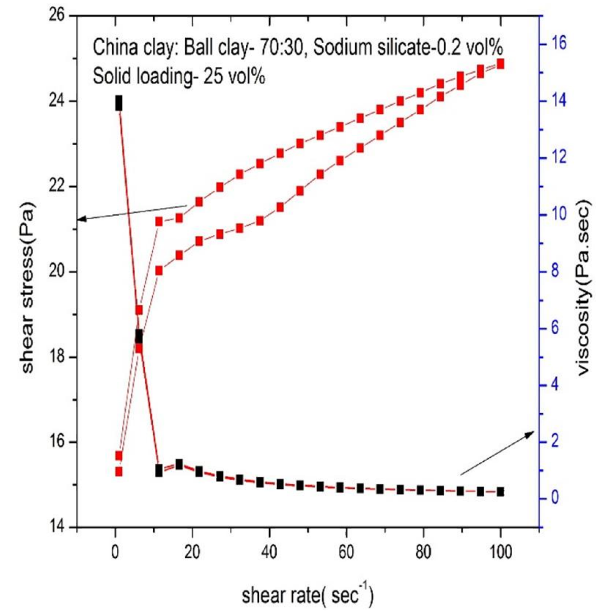
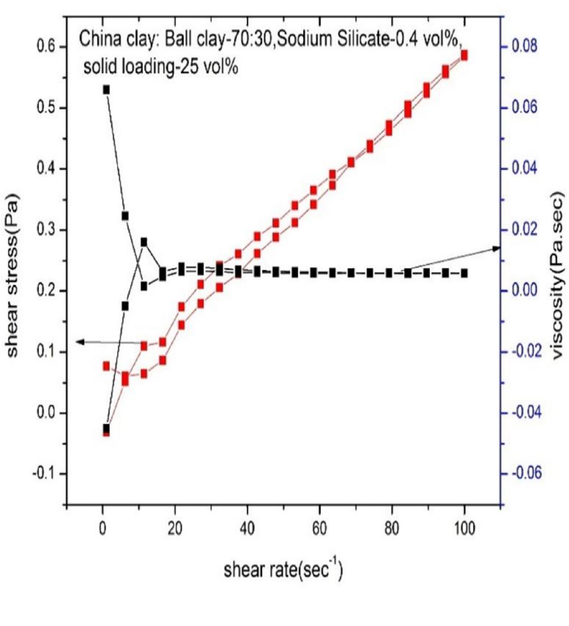


Figure 53: Variation of Shear Stress and Viscosity with Shear rate

Figure 54: Variation of Shear Stress and Viscosity with Shear rate



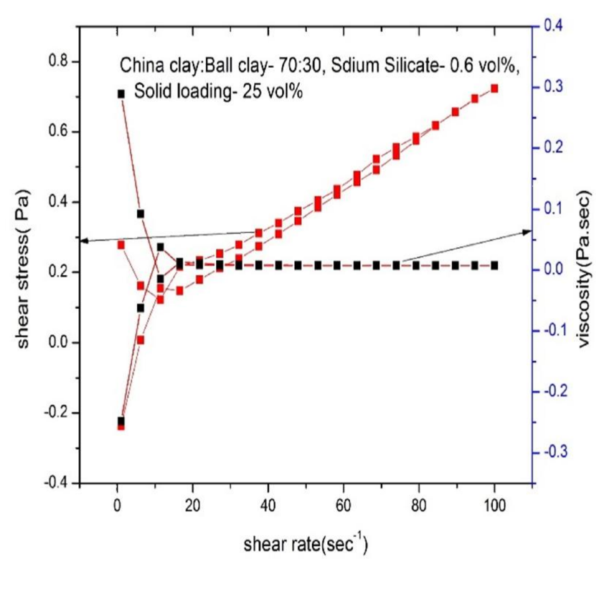


Figure 55: Variation of Shear Stress and Viscosity with Shear rate

Figure 56: Variation of Shear Stress and Viscosity with Shear rate

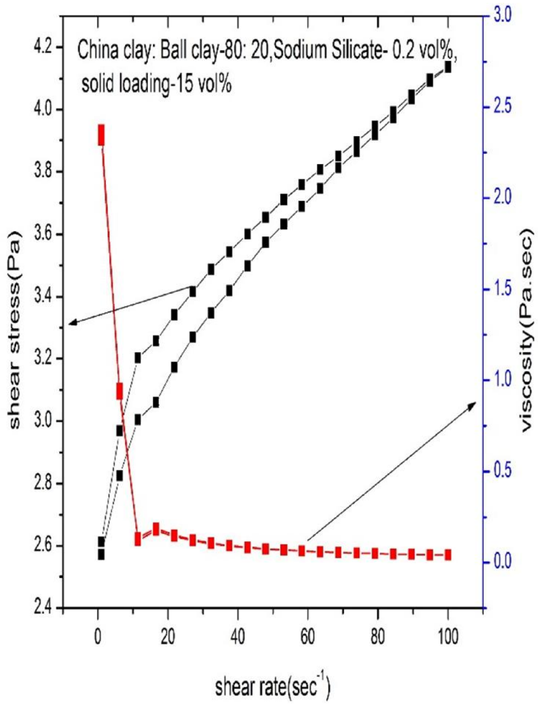


Figure 57: Variation of Shear Stress and Viscosity with Shear rate

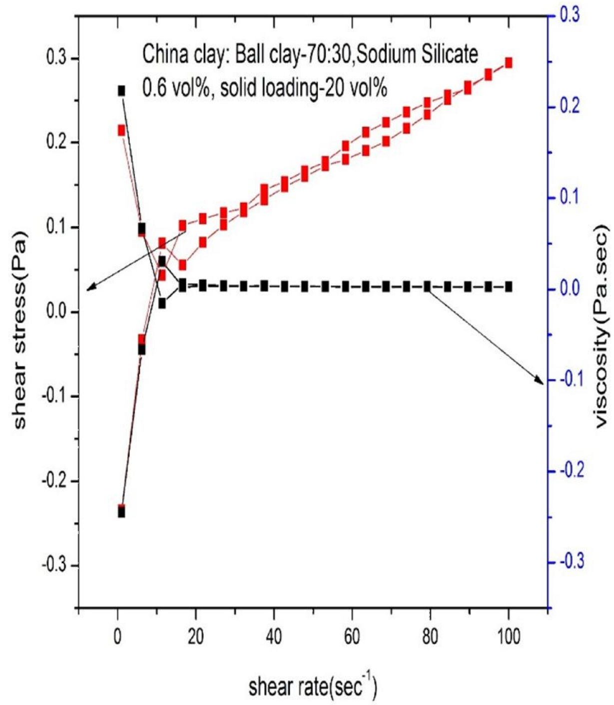


Figure 58: Variation of Shear Stress and Viscosity with Shear rate

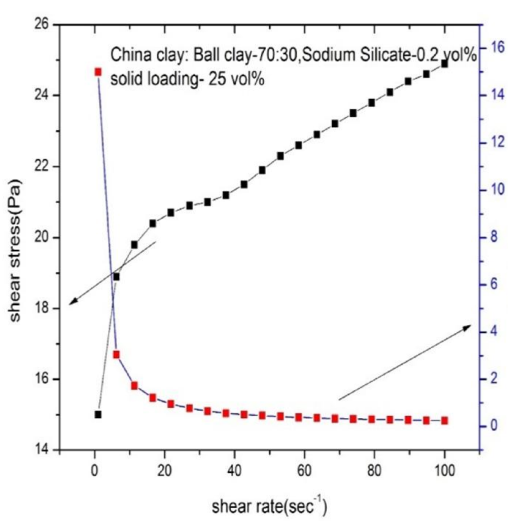


Figure 59: Variation of Shear Stress and Viscosity with Shear rate

4.1 Effect of solid loading on τ_{yield}, τ_{max} and τ_{gel} for varying amount of Sodium Silicate as deflocculant

Table.4- gives the variation of τ_{yield}, $\tau_{max,\ and}$ τ_{gel} for Sodium Silicate

China clay: Ball clay	Solid loading(vol%)	Deflocculant (vol%)	τ_{yield}(Pa)	τ_{max}(Pa)	τ_{gel}(Pa)
70:30	15	0.2	3.4	5.5	0.11
	20		9.3	16.7	0.36
	25		15	24.9	0.2
80:20	15	0.2	2.53	4.14	0.04
	20		3.81	6.54	0.7
	25		8.91	41.3	2.39
90:10	15	0.2	2.52	4.32	0.56
	20		3.43	5.94	0.07
	25		9.24	14	0.34
	15	0.4	0.0176	0.274	0.0771

4.2 Effect of solid loading on τ_{yield}, τ_{max}, τ_{gel} for varying amount of Darvan C as deflocculant

Table.5- gives the variation of τ_{yield}, $\tau_{max,}$ and τ_{gel} of slurries containing Darvan C

China clay: Ball clay	Solid loading(vol%)	Deflocculant(vol%)	τ_{yield}(Pa)	τ_{max}(Pa)	τ_{gel}(Pa)
70:30	25	0.2	7.62	22.1	3.88
80:20	20	0.2	2.58	5.68	0.43
	25		6.26	13.5	0.22
90:10	20	0.2	5.23	10.8	0.23
	25		6.1	14.7	0.67

4.3 Effect of solid loading on τ_{yield}, τ_{max}, τ_{gel} for varying amount of Sodium Carbonate as deflocculant

Table.6- gives the variation of τ_{yield}, $\tau_{max,}$ and τ_{gel} of slurries containing Sodium Carbonate

China Clay: Ball clay	Solid loading (wt%)	Deflocculant (wt%)	τ_{yield}(Pa)	τ_{max}(Pa)	τ_{gel}(Pa)
70:30	25	0.3	9.89	13.6	0.14
		0.4	5.97	7.54	0.22
		0.5	14.1	19.6	0.6

From table 4, 5and 6 it can be observed that for a particular solid loading with increase in deflocculant concentration ,the τ_{yield}, $\tau_{max,}$ and τ_{gel} increased.

4.4 Effect of solid loading on settling heights for varying amount of Sodium Silicate deflocculant

Table 7 gives the variation of settling heights of Sodium Silicate

China clay:Ball clay	Solid loading(vol%)	Deflocculant (vol%)	Difference in height(mm)	Difference in height/Initial height
70:30	15	0.2	5.12	0.191
		0.4	2.32	0.086
		0.6	5.4	0.26
	20	0.2	6.35	0.306
		0.4	5.28	0.167
		0.6	3.14	0.147
	25	0.2	10.98	0.55
		0.4	8.12	0.217
		0.6	9.64	0.315
80:20	15	0.2	2.28	0.124
		0.4	4.24	0.186
		0.6	2.16	0.118
	20	0.2	3.1	0.113
		0.4	2.58	0.138
		0.6	3.27	0.141
	25	0.2	11.08	0.607
		0.4	5.2	0.244
		0.6	1.08	0.055
90:10	15	0.2	2.44	0.109
		0.4	3.02	0.074
		0.6	3.22	0.077
	20	0.2	3.14	0.144
		0.4	6.68	0.308
		0.6	4.04	0.180
	25	0.2	3.08	0.171
		0.4	3.14	0.142
		0.6	2.96	0.151

4.5 Effect of solid loading on settling heights for varying amount of Darvan C deflocculant

Table 8 gives the variation of settling heights of slurries containing Darvan C

Table 8-

China clay:Ball clay	Solid loading(vol%)	Deflocculant (vol%)	Difference in height(mm)	Difference in height/Initial height
70:30	15	0.2	3.4	0.17
		0.4	4.98	0.173
		0.6	4.82	0.247
	20	0.2	9.92	0.357
		0.4	8.4	0.454
		0.6	4.66	0.223
	25	0.2	6.82	0.31
		0.4	6.94	0.36
		0.6	4.38	0.2
80:20	15	0.2	1.14	0.039
		0.4	12.08	0.522
		0.6	1.52	0.076
	20	0.2	3.72	0.116
		0.4	2.2	0.134
		0.6	0.22	0.134
	25	0.2	5.56	0.231
		0.4	1.92	0.097
		0.6	1.82	0.092
90:10	15	0.2	1.14	0.054
		0.4	5.92	0.251
		0.6	5.06	0.19
	20	0.2	1.64	0.072
		0.4	5.04	0.279
		0.6	6.56	0.284
	25	0.2	3.66	0.146
		0.4	2.82	0.121
		0.6	2.32	0.103

4.6 Effect of solid loading on settling heights for varying amount of Sodium Carbonate deflocculant

Table 9- gives the variation of settling heights of slurries containing Sodium Carbonate

Table 9-

China clay:Ball clay	Solid loading(vol%)	Deflocculant (vol%)	Difference in height(mm)	Difference in height/Initial height
70:30	25	0.3	5.6	0.45
		0.4	4.3	0.34
		0.5	6.4	0.56

4.7 Effect of solid loading on pH of slurry for varying amount of Sodium Silicate

Table 10 gives the variation of pH of slurries containing Sodium Silicate

China clay:Ball clay	Solid loading (vol%)	Deflocculant (vol%)	pH
70:30	15	0.2	9.28
		0.4	10.48
		0.6	11.19
	20	0.2	9.21
		0.4	10.33
		0.6	11.10
	25	0.2	8
		0.4	10
		0.6	11.31
80:20	15	0.2	9.37
		0.4	10.55
		0.6	10.92
	20	0.2	9.37
		0.4	10.14
		0.6	11.06
	25	0.2	8.54
		0.4	10.61
		0.6	11.38
90:10	15	0.2	9.48
		0.4	10.43
		0.6	11.08
	20	0.2	9.64
		0.4	10.28
		0.6	10.41
	25	0.2	9.37
		0.4	10.43
		0.6	11.55

4.8 Effect of solid loading on pH of slurry for varying amount of Darvan C

Table 11 gives variation of pH of slurries containing Darvan C

Table 11-

China clay:Ball clay	Solid loading (vol%)	Deflocculant (vol%)	pH
70:30	15	0.2	7.86
		0.4	7.64
		0.6	7.76
	20	0.2	7.74
		0.4	7.71
		0.6	7.46
	25	0.2	7.64
		0.4	7.74
		0.6	7.78
80:20	15	0.2	7.56
		0.4	7.70
		0.6	7.53
	20	0.2	7.81
		0.4	7.83
		0.6	7.86
	25	0.2	7.66
		0.4	7.6
		0.6	7.74
90:10	15	0.2	8.15
		0.4	7.9
		0.6	8.03
	20	0.2	7.82
		0.4	7.67
		0.6	7.83
	25	0.2	7.68
		0.4	7.53
		0.6	7.42

4.9 Effect of solid loading on pH of slurry for varying amount of Sodium Carbonate

Table 12 gives variation of pH of slurries containing Sodium Carbonate

Table 12-

China clay:Ball clay	Solid loading (vol%)	Deflocculant (vol%)	pH
70:30	25	0.3	10.85
		0.4	10.93
		0.5	11.03

From the above tables it can be observed that as the deflocculant concentration increased the pH of the slurries increased gradually.

4.10 Effect of variation of china clay and ball clay ratio on steady viscosity of slurry sample containing 0.2 vol% of Sodium Silicate

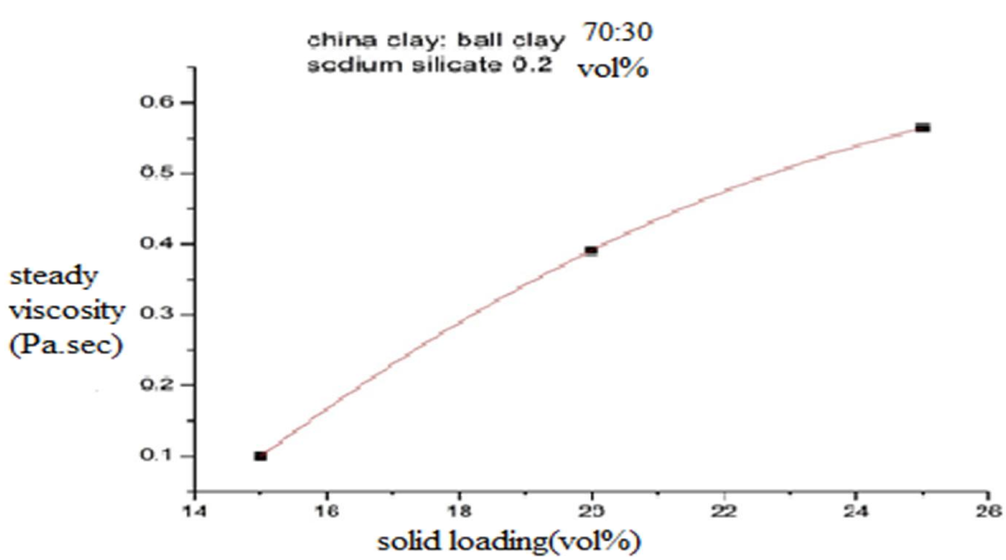
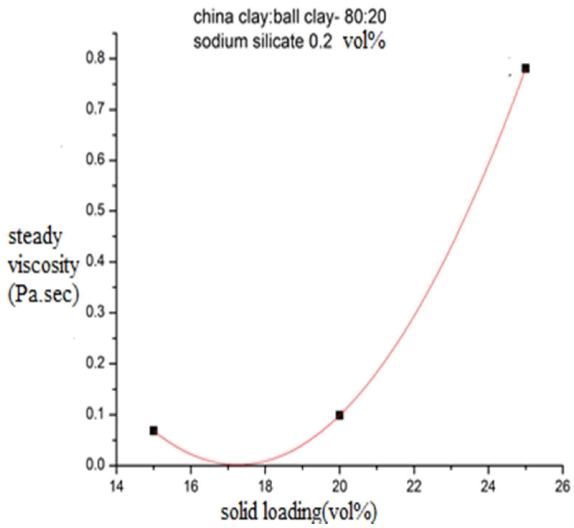


Fig 60: variation of steady viscosity with solid loading



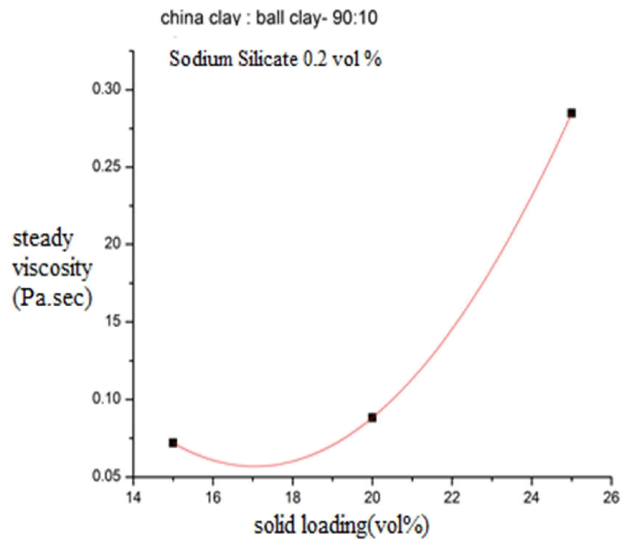
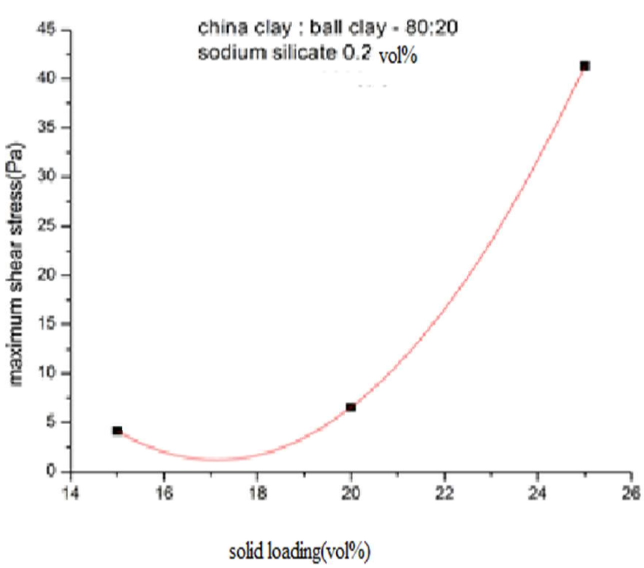


Fig 61: variation of steady viscosity with solid loading   Fig 62: variation of steady viscosity with solid loading

It can be observed from the graph that as the china clay proportion increased in the slurry, the steady viscosity increased for any solid loading. And with increase in solid loading the steady viscosity increased sharply from 20 vol% to 25 vol% than from 15 vol% to 20 vol% as the slurry became more viscous at higher solid loading.

4.11 Effect of variation of china clay and ball clay ratio on maximum shear stress of slurry sample containing 0.2 vol% of Sodium Silicate



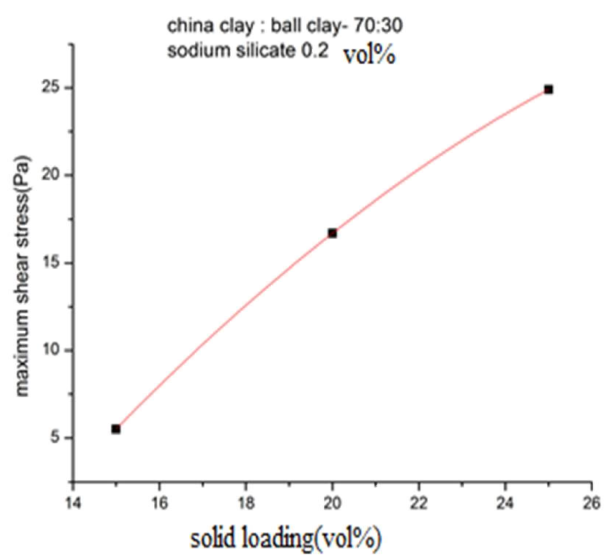


Fig 63: variation of maximum shear stress with solid loading   **Fig 64: variation of maximum shear stress with solid loading**

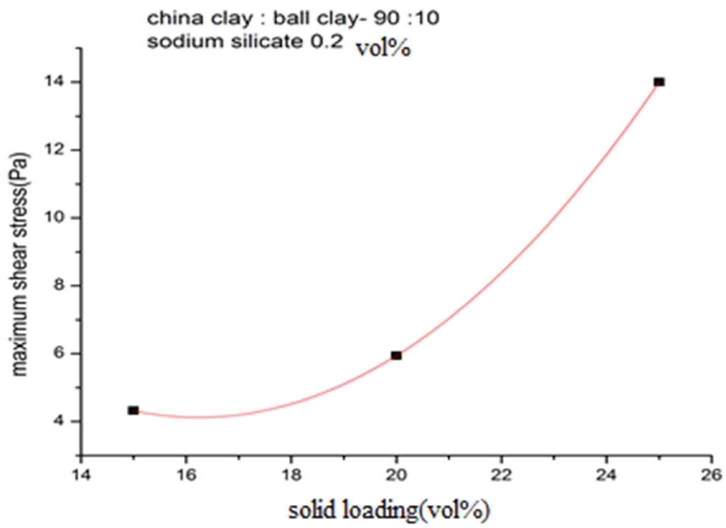


Fig 64: variation of maximum shear stress with solid loading

It can be noted from the graph that as the solid loading increased the maximum shear stress increased. And the extend of increasing the maximum shear stress with solid loading increased with china clay content.

4.12 Effect of variation of Sodium Carbonate concentration on steady viscosity

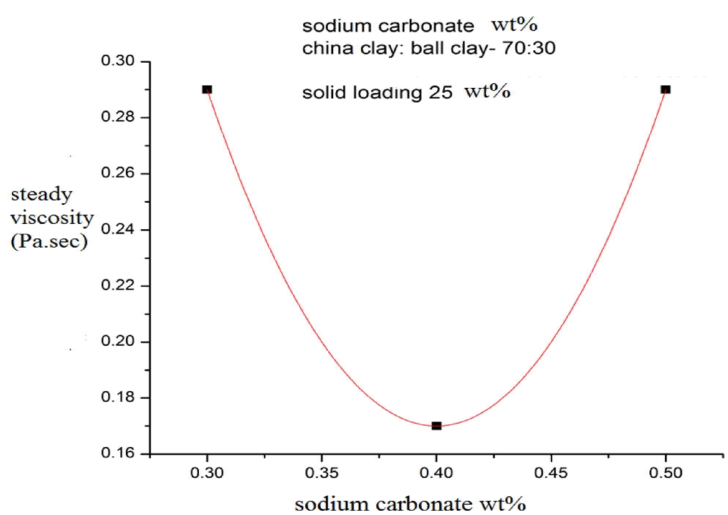


Fig 65: variation of steady viscosity with Sodium Carbonate content

From this graph it can be analyzed that with increase in Sodium Carbonate concentration in slurry sample the steady viscosity decreased and after getting optimized to a particular value again it increased to higher values.

CHAPTER 5

CONCLUSIONS

CONCLUSIONS

- With increase in china clay content in the slurry the viscosity of the slurry increased.
- With increase in deflocculant concentration, the viscosity decreased and getting optimized to a minimum value it increased.
- With increase in deflocculant concentration in slurries, the pH increased.
- The deflocculants (Sodium Silicate and Darvan C) should be added 0.2 vol% at maximum to a slurry containing any above composition of china clay and ball clay(70:30. 80:20, 90:10).
- Generally with increase in deflocculant concentration the pH of the slurry increased.

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